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(54) PARTICULATE DETERGENT COMPOSITION

(71) We, UNILEVER LIMITED, a company registered under the laws of Great Britain, of Port Sunlight, Birkenhead, Cheshire, England, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement: —

The present invention relates to detergent compositions and a process for preparing them. More particularly, the present invention relates to detergent compositions which contain enzymes.

It is known that enzymes, when incorporated in a particulate detergent composition, often lose their activity quickly during storage of the powder. Moreover, the dosing of the enzymes into the detergent powder and the homogeneous mixing of the enzymes with the detergent powder ofter presents problems, is it is not so easy to control the homogeneity of the powder.

It has now been found that these disadvantages can be significantly prevented by incorporating into the detergent composition discrete shaped solids in which enzymes are incorporated.

According to the invention a particulate detergent composition comprises particles of surface-active material and discrete, shaped inorganic solids containing enzymes. The material which forms the shaped solids consists of inorganic material with which the enzymes are compatible. The inorganic material should furthermore be readily soluble or dispersible in normal tap water at a temperature of about 20°C. The shaped solids may furthemore not be appreciably affected during storage of the detergent compositions.

Examples of suitable inorganic material are readily water-soluble or -dispersible inorganic salts such as sodium sulphate, sodium carbonate, sodium bicarbonate, acid ortho- and pyrophosphates such as sodium dihydrogenorthophosphate and acid sodium pyrophosphate, borates such as borax, boric acid, ammonium salts such as ammonium bicarbonate, ammonium chloride, and alkali metal triphosphates such as sodium triphosphate.

Furthermore, combinations of compounds which evolve a gas when brought into an aqueous solution, so-called effervescent substances, may be used to advantage in the present invention, thereby accelerating the disintegration of the shapel solids in the aqueous solution. Such combinations are, for example, combinations of acid salts or weak acids with carbonates or bicarbonates.

In general, the inorganic material should be solid at room temperature and should not be hygroscopic.

The enzymes which are incorporated into the discrete, shaped solids may be proteolytic, amylolytic and lipolytic enzymes and mixtures thereof. In partcular, proteolytic enzymes may be used, preferably of bacterial origin.

The amount of enzymes to be incorporated in the detergent composition is dependent upon the enzymatic activity of the particular enzyme used. In general, enzyme-containing discrete shaped solids are incorporated into the particular detergent composition in an amount such that the final product has an activity of 10^4—10^6 maltose units per kilogram of final product when amylolytic enzymes are used, and of 5—20 Anson units per kilogram of final product when proteolytic enzymes are used.

The discrete shaped solids may further contain other ingredients which are desirable in detergent compositions, provided they do not affect the enzymes adversely. They may e.g. contain compounds which stabilize or activate the enzymes. The shaped solids may also be coloured with a colouring agent, the discrete shaped solid being coloured on the surface or through-and-through, whereby a commercially attractive so-called speckled detergent is

obtained. The discrete shaped solids may have the same or different colours. The discrete shaped solids may also contain fluorescers.

The storage stability of the discrete shaped solids may be further improved by coating the discrete shaped solids with a suitable coating material. The coating material, which may also be coloured, should be sufficiently soluble or dispersible in normal tap water at a temperature of about 20°C. Examples of suitable coating material are nonionics, film-forming agents such as shellac. By proper selection of the ingredients of the shaped solid readily water-soluble or -dispersible solids can be obtained. Water-solubility may be increased by incorporating a hydrotrope into the shaped solid.

In general, the discrete shaped solids should dissolve or disperse or disintegrate in normal tap water at about 20°C within 10 minutes, preferably within 2 minutes. The pH of the shaped solids should preferably be the optimum pH for the enzyme used. In many cases the use of acid salts as exemplified before has a beneficial effect upon the stability of the enzymes.

The discrete shaped solids should have a physical form which is suitable for mixing with a particulate detergent composition. Suitable forms include ribbons, flakes, threads, small sphere, noodles, small tablets, pellets and granules. The shaped solids may be obtained by any suitable shaping process, such as milling, pelleting, extruding, stamping, pressing and granulation.

In general, the maximum dimension of a discrete shaped solid should be not more than 15 mm, and the minimum dimension should be not more than 2.5 mm. The discrete shaped solids preferably weight between 0.05 and 100 mg, particularly preferably between 2 and 20 mg.

For example, noodles with a length of 15 mm and a width of 0.5 mm, small tablets having a cross section of 2.5 mm and a thickness of 1.5 mm, flakes of 4 mm length and a thickness of 0.2 mm and pellets having a cross section of 2.5 mm are examples of suitable discrete shaped solids for use according to the invention.

Noodles and small tablets having a weight of about 20 mg are particularly suitable. Small tablets in which the inorganic material consists of a mixture of a weak acid or an acid salt with an alkali metal bicarbonate are in particular suitable. The weight ratio in which the enzymes and the inorganic material are mixed in the preparation of the discrete shaped solids lies between 1:100 and 100:1.

The particulate detergent composition into which the enzyme-containing discrete shaped inorganic solids are incorporated may be any suitable composition to which enzymes may be added. The composition normally comprises an active detergent, a water-soluble builder salt and normal detergent adjuvants. The active detergent may consist of an anionic, a nonionic, a cationic, an amphoteric detergent surfactant or a soap, or a mixture of these active detergents. The water-soluble builder may consist of organic and/or inorganic builder salts, such as alkaline metal condensed phosphates, alkaline silicates, alkali metal salts of ethylenediaminetetraacetic acid or nitrilotriacetic acids or mixtures of these builder salts.

The normal detergent adjuvants comprise bleaching agents such as sodium perborate, bleach precursors, stabilizers, corrosion inhibitors, germicides, solubilizers, perfumes, soil-suspending agents, fabric-damage inhibitors and colouring material.

These particulate detergent compositions may be brought into particulate form by any suitable process, such as spray-drying and mixing.

Although the present invention particularly relates to particulate detergent compositions, it is also possible to compress the particulate composition of the invention into a detergent tablet.

The detergent compositions of the invention show an improved enzyme stability during storage, and do not show a significant segregation of the discrete shaped solids in the composition during transport or storage.

The present invention will now be further illustrated by way of Examples, in which percentages are by weight.

EXAMPLE 1

Small tablets having a diameter of 2.5 mm, a thickness of 1 mm and weighing 7 mg were prepared by compressing 80% of an equimolecular mixture of sodium dihydrogenphosphate and sodium bicarbonate with 20% of a proteolytic enzyme (1.6 Anson units/g).

The rate of solubility in tap water of 20°C was 10 seconds.

A detergent powder containing 5% of these tablets did not show any significant segregation of the tablets in the composition. The loss of enzymatic activity during storage of the composition was significantly reduced.

EXAMPLE 2

Spherical granules having a diameter of 0.15 mm and weighing less than 0.5 mg were prepared on a rotating bowl from 75% of potassium dihydrogen phosphate, 20% of a proteolytic enzyme having a proteolytic activity of 1.6 Anson units/g and 5% of a nonionic having a melting point below 20°C.

EXAMPLE 3

Example 2 was repeated using as inorganic material acid sodium pyrophosphate, sodium dihydrogen orthophosphate and sodium triphosphate.

The rate of solubility in tap water at 20°C was less than 1 minute in all cases.

In the case of sodium dihydrogen phosphate as carrier material it is advantageous to use shellac as a coating material, since sodium dihydrogen orthophosphate may be converted in an alkaline detergent powder into disodium monohydrogen orthophosphate, which has a low melting point (about 30°C) and which may cause caking of the powder. Coating the sodium dihydrogen phosphate granules with shellac, dissolved in alcohol, may prevent this.

WHAT WE CLAIM IS:—

1. A particulate detergent composition comprising particles of surface-active material and discrete, shaped inorganic solids containing enzymes.

2. A detergent composition according to Claim 1, in which the discrete, shaped inorganic solids comprise a mixture of an acid salt or a weak acid with alkali metal carbonates and/or bicarbonates.

3. A detergent composition according to Claim 1 or Claim 2, in which the discrete, shaped inorganic solids weight between 0.05 and 100 mg.

4. A detergent composition according to Claim 3, in which the discrete, shaped inorganic solids weight between 2 and 20 mg.

5. A detergent composition as claimed in any one of Claims 1 to 4, in which the maximum dimension of the discrete, shaped inorganic solids is not more than 15 mm and the minimum dimension is not more than 2.5 mm.

6. A detergent composition as claimed in any one of Claims 1 to 5, in which the enzymes are amylolytic, proteolytic or lipolytic enzymes.

7. A detergent composition according to any one of Claims 1 to 6, in which the discrete, shaped inorganic solids are coated with a water-soluble or water-dispersible coating material.

8. A detergent composition according to any one of Claims 1 to 7, in which the discrete, shaped inorganic solids are coloured.

9. A detergent composition according to Claim 8, in which the discrete, shaped inorganic solids are coloured with different colours.

10. A detergent composition substantially as herein described with reference to the Examples.

11. A detergent tablet comprising a compressed detergent composition according to any one of the preceding claims.

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